Processing, Microstructure and Properties of Micro and Nano Alumina Reinforced Aluminium Composites

THIS THESIS IS SUBMITTED IN THE PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF **BACHELOR OF TECHNOLOGY**

IN

METALLURGICAL AND MATERIALS ENGINEERING

BY

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA
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CERTIFICATE

This is to certify that the thesis entitled **"Processing, Microstructure and Properties of Micro and Nano Alumina Reinforced Aluminium Composites"** submitted by **Saurendra Nag (109MM0082)** and **Neeradh Talluri (109MM0657)** in partial fulfillment of the requirements for the award of **BACHELOR OF TECHNOLOGY** degree in **Metallurgical and Material Engineering** at **National Institute of Technology, Rourkela** is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

Date: 8th May, 2013

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Date: 8th May, 2013

Place: Rourkela

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Abstract

The evolution of microstructure by varying the particle size of reinforcement in the matrix by employing powder metallurgy has been demonstrated here in Al–Al_2O_3 system. An emphasis has been laid on varying the reinforcement particle size and evaluating the microstructural morphologies and their implications on mechanical performance of the composites. Nano composites of 8 volume % alumina (average size < 50 nm) reinforced in aluminium matrix were fabricated by powder metallurgy route. Another set of specimens having same composition (average size ~ 10 μm) had been fabricated to compare the physical as well as mechanical attributes of the microcomposites as well as the nanocomposites. These micro- and nano-composites have been characterized using X-ray diffraction, scanning electron microscopy and followed by density, microhardness, compressive strength, wear resistance and corrosion resistance measurements. The alumina nanoparticles revealed an interface showing appreciable physical intimacy with the aluminium matrix compared to that of the alumina microparticles. By employing the powder metallurgy route the agglomeration is reduced and no new phases were formed as the temperatue is very low.

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CHAPTER 1

INTRODUCTION

1. Introduction

Al and Al alloys became attractive candidate for the application in aerospace, defence and automotive industries owing to their versatile properties. These applications require high strength with low density. The strength of Al and Al alloys can be enhanced by heat treatment, thermo mechanical processing, alloying addition, severe plastic deformation and so on. However, all these processes have their own drawbacks. The development of metal matrix composite is a innovative idea for enhancing the strength of Al and Al alloys. In this regard, the aluminum metal matrix composites (AMMCs) received particular attention in the automobile and aerospace industries as potential advanced structural materials in the past three decades owing to their

- High specific strength
- Improved stiffness
- Reduced density
- Improved high temperature properties (creep, oxidation, corrosion)
- Control thermal expansion coefficient
- Improved abrasion and wear resistance

The composites reinforced with long continuous fibers are expensive; the short fibers reinforced composites show anisotropic properties; the whiskers causes health hazard. Therefore, the particle reinforced composites have to be developed for such applications. The particle reinforced composites are economic and having isotropic properties, low density and easy to fabricate.

There have been several literatures on the micro particles reinforced aluminium matrix composites. However, there are very few reports exist on the nano particles reinforced aluminium matrix composites although the nano particles reinforced composites are expected to exhibit the superior properties [4]. Most of the literatures on the micro and nano reinforced

AMMCs focused on the mechanical behaviour only [6]. In addition, there are very few literatures that compared the performance of micro particles reinforced AMMCs with that of the nano particles reinforced AMMCs [4]. However, the volume fraction of the micro and nano particles in the composites were not kept the same, which is very much require for the valid comparison. Furthermore, the wear and corrosion behaviour of the micro and nano particles reinforced AMMCs is not yet properly understood. Therefore, in the present investigation the mechanical properties along with wear and corrosion performance of the micro and nano particles reinforced pure Al matrix composites were evaluated.

The objective of the present investigation is to evaluate the mechanical properties along with wear and corrosion performance of the micro and nano particles reinforced pure Al matrix composites. The composites were processed via powder metallurgy route with conventional sintering. The various combinations used for the composites are as follows:

Sr. No.	Composition used	Nomenclature used
1	Al + 8% Nano Al_2O_3	Al+NA
2	Al+ 8% Micro Al_2O_3	Al+MA

CHAPTER 2

LITERATURE REVIEW

2. Literature review

In order to gain background knowledge on the previous work done in similar fields, various papers and journals were studied. The findings of some of the journals are enumerated below.

Hsabi et al. [2] investigated the compressibility of aluminium/alumina reinforced nano composite by blending and mechanical milling. The compressibility of the blended and milled aluminum/nano scale alumina particles exhibits the same features as the typical metal powder compaction. The consolidation is mainly generated by two phenomena: particle rearrangement and plastic deformation. The addition of hard nanoparticles to aluminum powder by mixing improves the densification capacity at particle rearrangement due to disintegration of the clusters and agglomerates under the applied load and filling the voids between the matrix particles.

Poirier et al. [1] proposed mechanical milling as a possible way to disperse uniformly nano-Al_2O_3 in Al powder. This milling helped in uniform dispersion of the second phase with few agglomerates of around 1micron in size. Also, the hardness of Al-Al_2O_3 nanocomposite is nearly five times greater than pure unmilled aluminium. Observed strengthening is due to grain refinement and dispersoid formation from mechanical milling.

Rahimian et al. [11] studied the effect of particle routesize and amount of reinforcement on the microstructure and mechanical properties of aluminium matrix composites made by powder metallurgy. Decreasing the alumina particle size increases the hardness. Furthermore, as the alumina amount decreases in the composite, grain size and alumina particle distribution homogeneity increases. Alumina additions reduce the wear rate of the composite. The finer particle size of alumina presents greater yield and compressive strength.

Mazahery et al. [3] studied nanosized Al_2O_3 particle-reinforced aluminum matrix composites when fabricated by stir casting, using the mixture of nanosized Al_2O_3 and microsized Al particles as reinforcement. Microstructural observations revealed a reasonably uniform distribution of Al_2O_3 nanoparticles in the Al matrix. These particles refined the grain structure of the cast

materials. There are also a few agglomerated particles through the matrix. The addition of nanoparticles resulted in significant improvements in both compressive and tensile flow stress.

Dash et al. [4] studied the synthesis and characterization of aluminium – alumina micro and nano composites by plasma spark sintering Al–Al_2O_3 nano composites with 0.5, 1, 3, 5, 7 vol.% alumina nanoparticles and micro composites with 1, 5, 20 vol.% alumina micron size particles were fabricated by spark plasma sintering method. The distribution of alumina particles in the aluminium matrix is homogeneous and uniform both in nanocomposites and microcomposites (slightly better distribution in nanocomposites than microcomposite). The interface of aluminium and alumina in nanocomposites is seemingly sound than in the case of microcomposite i.e. the compatibility of alumina in aluminium matrix in nanocomposites is better than in the microcomposites. The density of microcomposites as well as nanocomposites decreases with increasing alumina content.

Zabihi et al. [5] investigated aluminum/alumina metal matrix composite strips when produced by powder metallurgy and the hot rolling processes have a good bonding quality between Al and Al_2O_3 particles . Redistribution of alumina particles in aluminium matrix is improved by hot rolling process. Tensile strength and hardness increases with increase in alumina content in the matrix and the percentage of elongation decreases.

CHAPTER 3

EXPERIMENTAL PROCEDURE

3. Experimental procedure

3.1. Materials: In the present investigation the commercially pure Al is reinforced with the micro and nano alumina particles. The various combinations used for the composites along with the nomenclature used are shown in Table 3.1.

Table.3.1. Table showing the composition and nomenclature used for the composites

Sr. No.	Composition used	Nomenclature used
1	Al + 8% Nano Al_2O_3	Al+NA
2	Al+ 8% Micro Al_2O_3	Al+MA

3.2.Compaction

As received aluminium and alumina powders of micro and nano scale of 20g were mixed such that the volume fractions of alumina in the mixtures were 8% in both micro and nano scale. Six samples of same weight were prepared with three for each scale. Then the samples were taken and blended together properly using an abrasion tester and were allowed for 2 revolutions to blend uniformly. The powders were taken in a plastic container along with few steel balls to ensure uniform distribution of the alumina particles throughout the aluminium matrix. The blended samples were then cold compacted by applying a load of 600 MPa in a die of 15 mm diameter. A lubricant (Zinc-Sterate) was used to ensure the easy removal of the compacted cylindrical sample.

3.3. Sintering

The compacted pellets were taken and heated in a tubular furnace in an inert atmosphere (99.99% pure argon gas) at temperatures of 600°C for a holding time of 2 hours to ensure the densification of the compacted powder samples. The densities of the sintered samples were calculated and noted.

3.4. Density measurement

The theoretical, green and sinter density of the samples were calculated using rule of mixtures.

Theoretical Density

$$D_c = D_m * V_m + D_f * V_f$$

Where D_c, D_m, D_f – densities of the composite, matrix and reinforced phase respectively;

V_m,V_f – volume fraction of the matrix and reinforced phase respectively.

Green Density

D_{gc} = *Mass of the compacted composite (m) / Volume of the compacted composite (V)*

$V = \pi d^2 h/4$

Where d = Diameter of the compact

h = height of the compact

3.5. X-ray diffraction analysis

The XRD analysis was carried out for all the composites using PANnalytical X-ray Diffractometer. The 2θ angle was varied from 10° to 90° and the scanning rate used was 2° per minute.

Fig.3.1. X-ray Diffractometer

3.6. SEM analysis

Microstructural characterization using SEM was done to observe the microstructure for all the composites in as sintered condition and after the wear test. The Al-Al_2O_3 samples were mechanically polished using standard metallographic techniques before the examination.

- Samples were polished using emery paper of designation 1/0, 2/0, 3/0, and 4/0 respectively changing the direction of polishing about 90° after every operation under a given paper for efficient removal of scratches on the surface.

- Cloth polishing was done in order to remove fine scratches which were not removed during paper polishing.

Etching was done using the Keller's reagent (1% HF (48%), 1.5% HCl, 2.5% HNO_3 and 95%water). The SEM micrographs of the samples were obtained. The images were taken in secondary electron (SE) mode.

Fig.3.2. Scanning electron microscope

3.7. Hardness measurement

The hardness of the composite specimens was measured by Vickers hardness tester (LECO-LV700). Micro hardness of all the samples was measured under a load of 100 gm for a dwell time of 10 seconds. As a result of the indenter's shape, the impression on the surface of the specimen was a square. The length of the diagonals of the square was measured through a microscope fitted with an ocular micrometer. Machine itself displays the hardness value in

Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH). A minimum of 3 readings were taken on each sample and checked for consistency.

Fig.3.3. Vickers Micro hardness testing machine

3.8. Wear testing

Wear behavior of composite specimens has been studied on 'pin-on disc type' wear testing instrument (Model: DUCOM TR-20-M100) having a steel disc of diameter 50 mm with a hardness of 64 HRC. A required normal load was applied through a lever mechanism when the samples were held stationary. The tests were carried out by varying one of the following three parameters and keeping other two constants:

- Applied load is 30 N
- Linear velocity 1.57m/s
- Sliding distance is 1000 m

Test is carried out in dry conditions. Uniformity in experiential procedure can be achieved by cleaning the disc with emery paper and small amount of water to avoid the entrapment of wear debris. Scanning electron microscopy was used to analyze the morphology of the worn surfaces of sample.

Fig. 3.4. Pin-on-disc wear and friction monitor machine

Fig.3.5. Full view of Ducom Wear and Friction Monitor

The wear loss is then calculated using the following expression

Wear rate = Volume loss / Sliding Distance (mm^3/m)

3.9. Compression test

The compressive strength of the composite was analyzed using a universal testing machine (Instron-SATEC series servo-hydraulic machine) at a crosshead speed of 0.5mm/min. The specimens were reduced to 50% from their original dimension and stress at 50% reduction was measured. A plot was drawn comparing stress at 50% reduction and the samples with micro and nano reinforcement particles.

Fig.3.6. Universal Testing Machine

3.10. Corrosion test

Corrosion tests were performed on all the specimens after slightly grinding the surfaces with silicon carbide paper (2500 grit, deionized water cooling). Finally, all the specimens were cleaned in alcohol prior to corrosion test. Electrochemical corrosions tests were carried out in aqueous 3.5 wt.% NaCl solution saturated with atmospheric oxygen and adjusted to neutral pH using NaOH with an exposed area of 0.5 cm^2 using a Gill AC potentiostat/galvanostat. The corrosion cell (333 ml) with a typical three electrode set-up, with the specimen as the working electrode, a saturated Ag/AgCl electrode as reference electrode and a platinum mesh as counter electrode was used. The electrolyte temperature was controlled at 22± 0.5°C and the electrolyte was stirred during the experiments. The experiment consisted of Potentiodynamic polarization scan starting from −150 mV relative to the free corrosion potential with a scan rate of 0.2 mV/s. The test was terminated when a corrosion current density of 0.1 mA/cm^2 was exceeded to minimize the damage on the specimen surface. This test lasted for about 30 minutes. From the cathodic and anodic branch of the polarization curve the corrosion rate was determined using the Tafel slope.

CHAPTER 4

RESULTS AND DISCUSSIONS

4. Results and discussion

4.1. Porosity

Porosity = (Theoretical Density – Experimental Density)

4.1.1. Theoretical Density:

For Micro particle reinforced sample, $D_m(Al) = 2.7$ g/cc and $D_f(Al_2O_{3(m)}) = 3.95$ g/cc and the volume fraction of the reinforcement phase is $V_f = 8\%$

Now $D_c = 2.7*0.92 + 3.95*0.08$

$D_c = 2.8$ g/cc

For Nano particle reinforced sample $D_m(Al) = 2.7$ g/cc and $D_f(Al_2O_{3(n)}) = 3.97$ g/cc and the volume fraction of the reinforcement phase $V_f = 8\%$

Now $D_c = 2.7*0.92 + 3.97*0.08$

$D_c = 2.8016$ g/cc

4.1.2 Green Density:

For Micro particle reinforced samples,

Table.4.1. Table showing the Green densities of the micro particle reinforced samples (Al+MA)

Sample No.	Mass (g)	Diameter(mm)	Height(mm)	Volume(cm^3)	Density(g/cc)
1	5.807	14.17	13.128	2.078	2.805
2	5.757	14.166	13.117	2.067	2.785
3	5.805	14.164	13.128	2.068	2.806

For Nano particle reinforced samples,

Table.4.2 . Table showing the Green densities of the nano particle reinforced samples (Al+NA)

Sample No.	Mass (g)	Diameter(mm)	Height(mm)	Volume(cm^3)	Density(g/cc)
1	5.786	14.167	13.137	2.07	2.7950
2	5.812	14.178	13.148	2.075	2.7999
3	4.851	14.17	11.128	1.754	2.7692

4.1.3 Sinter Density: Sinter density is measured from the mass of the composite and volume of the composite after sintering

For Micro particle reinforced sample

Table.4.3. Table showing the Sinter densities of the micro particle reinforced samples (Al+MA)

Sample No.	Mass (g)	Diameter(mm)	Height(mm)	Volume(cm^3)	Density(g/cc)
1	5.802	14.174	13.142	2.073	2.797
2	5.751	14.172	13.129	2.071	2.776
3	5.799	14.17	13.132	2.070	2.80

For Nano particle reinforced sample

Table.4.4. Table showing the Sinter densities of the nano particle reinforced samples (Al+NA)

Sample No.	Mass (g)	Diameter(mm)	Height(mm)	Volume(cm^3)	Density(g/cc)
1	5.774	14.18	13.149	2.076	2.78
2	5.806	14.176	13.169	2.078	2.79
3	4.842	14.177	11.101	1.752	2.76

Now porosity

Porosity = Theoretical Density – Experimental Density

Table.4.5. Table comparing the % porosity of micro and nano composite

Sample	Experimental Density	Theoretical Density	% Porosity
Al+MA	2.7986	2.8	0.14
Al+NA	2.788	2.8016	1.3

From the above results we can observe that nano composite has higher porosity than the micro composite. There is a difference between the theoretical and calculated density. This means porosity is present, which is the natural characteristic of materials processed by powder metallurgy route.

4.2 Microstructure:

Microstructure analysis is done under a scanning electron microscope. Both the secondary electron image and the back scattered images were taken for both micro and nano composites. The obtained micrographs are as follows

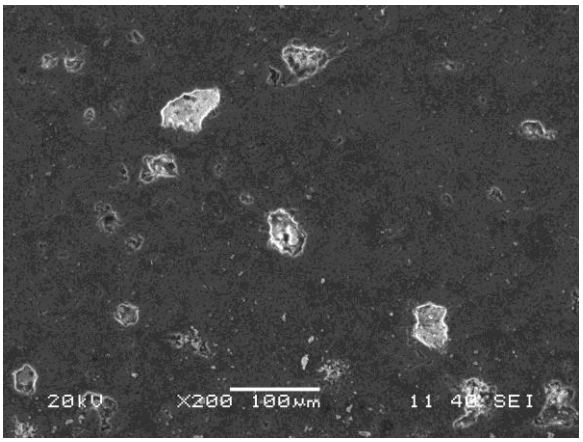


Fig.4.1.SEM (SE) image for Al+MA

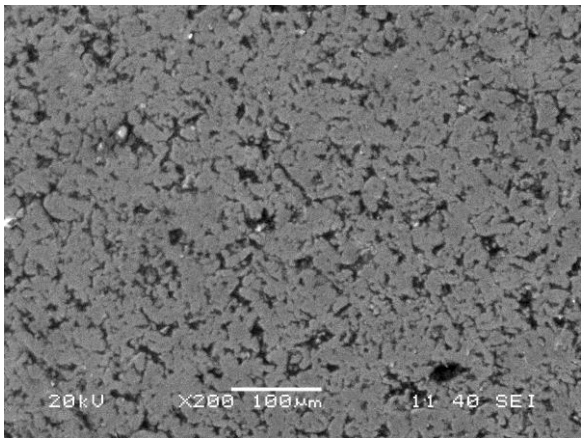


Fig.4.2. SEM (SE) image for Al+NA

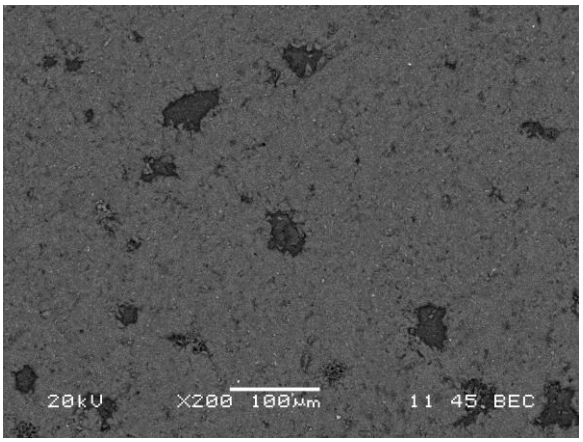


Fig.4.3. BSE image for Al+MA

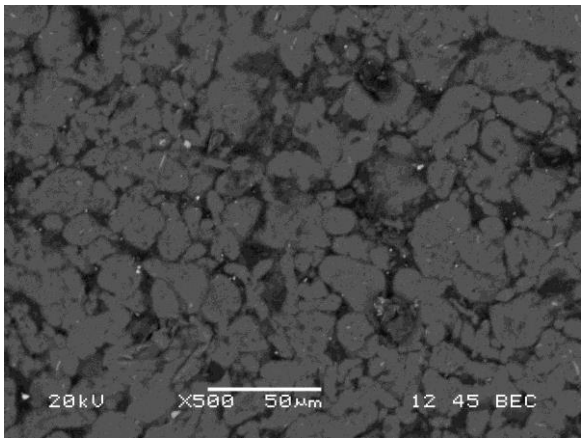
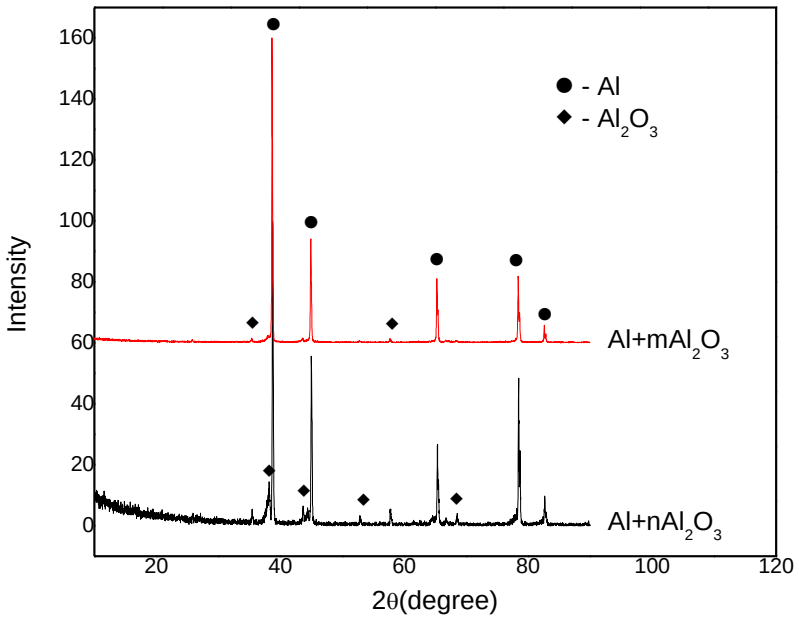


Fig.4.4. BSE image for Al+NA

Form the Fig. 4.1 and 4.2 it is observed that the composites exhibited good interface bonding between the particles and the matrix. It is also observed that there is an uniform distribution of nano and micro reinforcement particles in the aluminium matrix. No agglomeration of the nano particles is observed in the micrographs. The liquid metallurgy route generally causes agglomeration of nano particles. However, in the present investigation negligible agglomeration was observed because of the use of the powder metallurgy technique. No reaction products are seen at the interface of particles and matrix. By observing the figures 4.2 and 4.4 we can see that grain refinement has taken place.

4.3 X-ray diffraction analysis

The X-ray diffraction pattern obtained from the sintered samples of micro and nano composites is given as follows



The XRD analysis shows the peaks corresponding to pure aluminium and alumina. No other phases are formed.

4.4 Hardness measurement

The hardness measurement was done using a Vickers micro hardness tester (LECO LV700) and 9 measurements were taken for each sample to reduce the error and the obtained values are cited below in the table.

Table.4.6. Microhardness values for both micro and nano composite

Sample	1	2	3	4	5	6	7	8	9	Average hardness
Al+mAl_2O_3	39.1	37.7	39.6	34.1	32.9	36.0	33.6	35.0	36.0	36±2.38
Al+nAl_2O_3	39.0	40.6	38.9	40.8	37.0	42.2	42.7	41.9	40.4	40.38±1.82

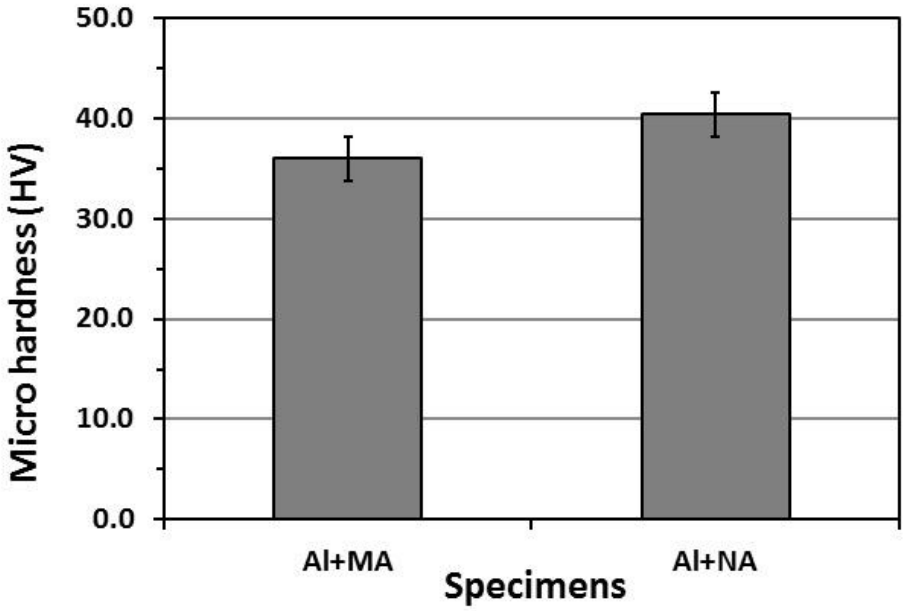


Fig.4.6 . Plot comparing the microhardness of micro and nano composite

From the figure 4.6 and table 4.6 we can observe that microhardness of nano composite is higher than that of micro composite, this might be due to the the Orowan strengthening mechanism and grain refinement. The Orowan strengthening can be explained by the expression

$$\tau = Gb/l$$

Where τ= Stress required to let the dislocation by pass the precipitate particles

G= Shear modulus of the matrix

b = Burgers vector of the dislocation

l= Distance between the precipitate particles

As the distance between the precipitate particles decreases the stress required to let the dislocation by pass increases which implies that hardness is higher if the distance between the precipitate particles is smaller.

The grain refinement can be explained by the Hall – Petch equation

$$\sigma_y = \sigma_i + kD^{-1/2}$$

Where, σ_y = Yield stress

σ_i = Friction stress

K = Locking parameter (constant)

D = Grain diameter

4.5 Compressive strength

The stress values withstand by all the composites at their 50% reduction of the specimens height are reported here.

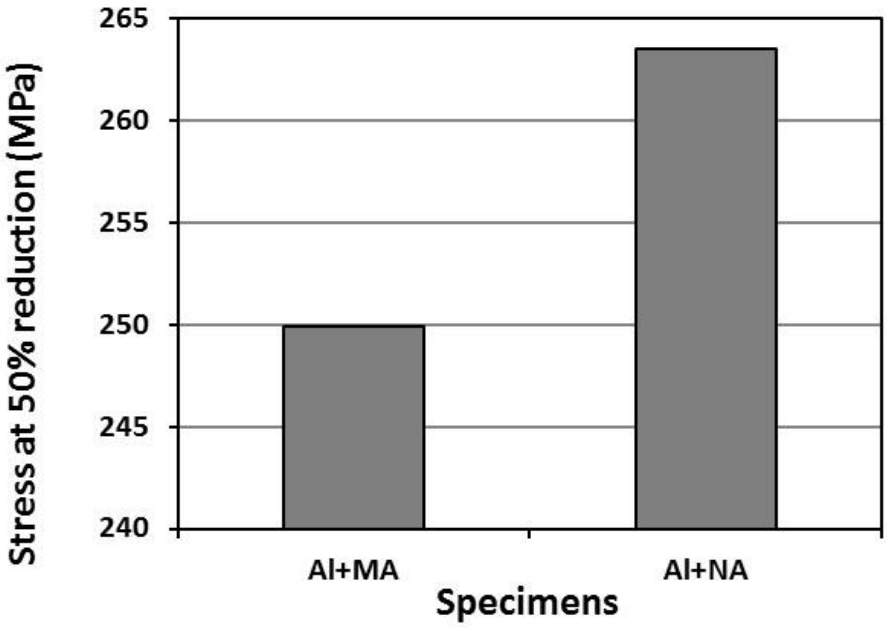


Fig.4.7. Plot comparing the stress at 50% reduction between micro and nano composites

The stress obtained is not the ultimate compressive stress but the stress at which the material is reduced to 50%. From the plot we can observe that the stress value for 50% reduction of the nano composite is greater than that of the micro composite. This is probably due to the Orowan strengthening and grain refinement observed on the nano composites.

4.6. Dry sliding wear behaviour

The dry sliding wear results shows that the wear rate is higher for the nano composite as compared to the micro composites. The increase in wear resistance of the Al+NA composites is due to the higher hardness values of the same.

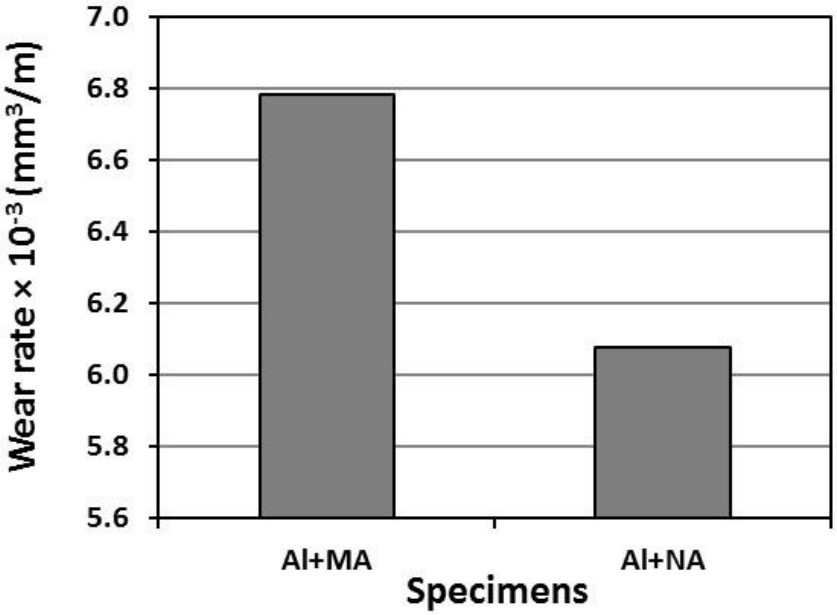


Fig.4.8. Plot comparing the wear rate between micro and nano composites

After completion of the wear tests the worn surfaces were observed under SEM to understand the underlying wear mechanisms. The representative micrographs obtained are as shown in Fig. 4.9 and Fig. 4.10 for the micro and nano composites, respectively.

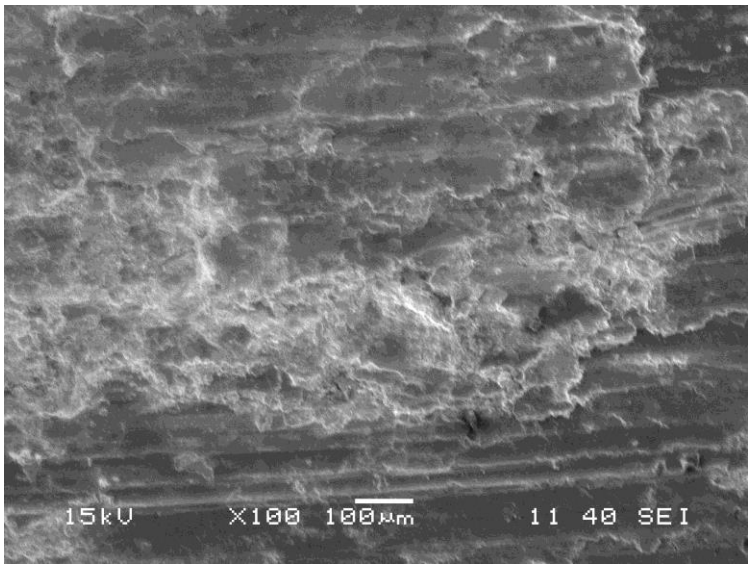


Fig.4.9. Micrograph showing the surface of micro composite

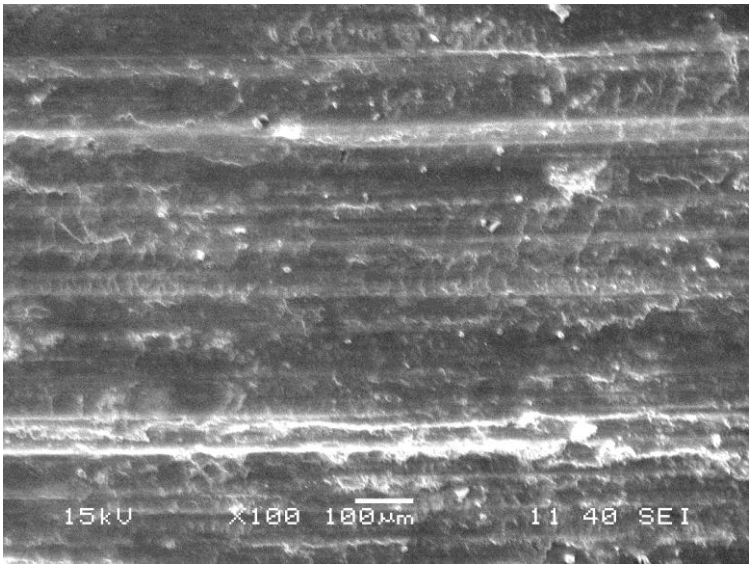


Fig.4.10. Micrograph showing the surface of nano composite

Form the figure it is clear that ploughing has taken place on the surfaces of the both micro and nano composites. The ploughing is associated with the wear mechanism abrasion. Although abrasion was observed in both cases, however, the severity of the abrasion was comparatively less in case of the nano composite. The improved wear resistance of the nano composites is owing to the higher hardness as compared to that of the micro composite.

4.7 Corrosion test

Electrochemical corrosion test was conducted on all the composites and the corrosion rate calculated from the potentiodynamic polarization plot is shown in Fig. 4.12.

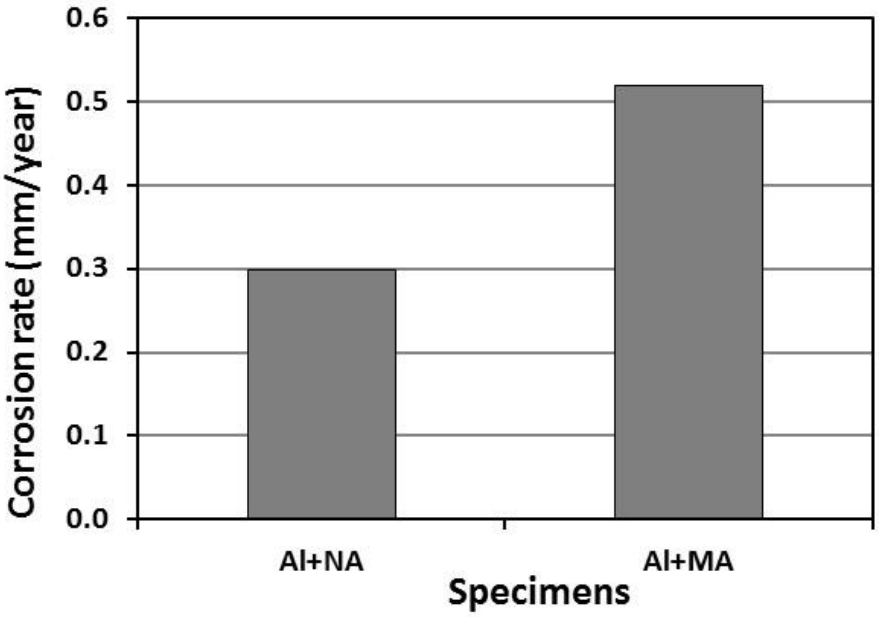


Fig.4.12. Plot comparing the corrosion rate between micro and nano composite

From the figure 4.12 it is observed that the nano composite exhibited better corrosion resistance as compared to that of the micro composites. The improved corrosion resistance of the nano composites is probably owing to the grain refinement observed for it. However, the corrosion mechanism for composite in general is not yet fully understood and need to be investigated in detailed in future.

CHAPTER 5

CONCLUSION

5. Conclusions

In the present investigation, the mechanical properties along with wear and corrosion performances of the micro and nano particles reinforced Al matrix composites were investigated. The composites were fabricated via powder metallurgy route. The following are the conclusions drawn from the present study.

- Fabrication is done successfully and seems to be alright with reasonable porosity
- Nano composite exhibited better property than that of the micro composites. The strength and hardness was higher for the nano composite probably due to the Orowan strengthening and grain refinement observed in the nano composite.
- Wear resistance was better in the nano composite because of its higher hardness.
- Corrosion resistance of the nano composite is better than that of the micro composite and this probably due to the grain refinement observed in the nano composites..

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